PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Method for the Preparation of Isoprene

We, COMPAGNIE DE PRODUITS CHIMIQUES ET ELECTROMETALLURGIQUES ALAIS, FROGES & CAMARGUE, of 23, rue de Balzac, Paris, France, a body corporate organized under French law, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement :—

The preparation of isoprene by a simple reaction having an excellent yield presents a considerable industrial importance.

We have found that this preparation can be effected very conveniently by starting from gamma isoamylene methyl ether (1-methoxy-3-methyl butylene-4), by the reaction :

$$CH_2=C(CH_3)-CH_2-CH_2-OCH_3 \longrightarrow CH_2=C(CH_3)-CH=CH_2+CH_3OH.$$

According to the invention, this reaction is effected in the vapour phase under a pressure equal to or lower than the atmospheric pressure, in presence of alumina or silicate of aluminium. These dehydration catalysts can be improved and in particular their activity can be prolonged by the addition of phosphate of copper or sulphate of magnesium, lead or barium. The reaction takes place within a wide range of temperature, but preferably in the vicinity of 300° C., and is accompanied by a dehydration of the methanol formed, which is converted into di-methyl ether.

EXAMPLE

Gamma-isoamylene methyl ether (prepared as indicated in our pending Application No. 15764 (Serial No. 663,117), filed June 14th 1949) is introduced by the aid of a capillary tube at a speed of 500 grammes per hour into an oven heated at 310° C., containing 1 litre of catalyst composed of kaolin with 5% by weight of copper phosphate, and maintained under a vacuum of 60 mm. of mercury. The catalyst is employed in the form of small cylinders (diameter about 3 mm., length 10 to 20 mm.). In order to obtain the catalyst in this form, finely powdered kaolin and ground copper phosphate are mixed with water to a pasty consistency ; the paste is thereafter forced through a calibrated tube and it is dried in air at the temperature of 20° C. until hardened. The oven, electrically heated, is slightly inclined, and the gamma-isoamylene methyl ether is introduced at the upper part. The gases leaving the oven are dried in a column filled with anhydrous calcium chloride, condensed in a receiver cooled by liquid nitrogen, and subjected to fractional distillation.

There are thus collected, in theoretical quantity, isoprene and di-methyl ether, this last resulting from the secondary reaction :

$$2\,CH_3OH=CH_3OCH_3+H_2O$$

which takes place in contact with kaolin, at the working temperature.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is :—

A method for the preparation of isoprene, consisting in passing 1-methoxy-3-methyl butylene-4, in the vapour phase, at elevated temperature, over a dehydration catalyst as hereinbefore defined.

Dated the 28th day of June, 1949.

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